

REMARKS

Claims 1 to 16 are pending. Claim 16 has been withdrawn from consideration. Claims 1, 2, 7, 12, and 14 - 16 have been amended. Claims 17 and 18 have been added. Claim 16 has been withdrawn because of a restriction requirement. The amendments to the specification are to correct typographical and/or grammatical errors. Bases for the claim amendments are in the specification at page 3, lines 4 and 26-28; pages 4-5, page 11; and the examples.

The provisional election of Examiner's claim group I (claims 1-15) with traverse is confirmed, and reconsideration is respectfully requested. It is urged that claim 16 is linked to claims 1-15, in that it is drawn to a method of finishing a surface (such as a car) utilizing a finishing composition as described in claims 1-15. It is submitted that a search for the finishing compositions of claim group I will, of necessity, include a search of those classes in which claim 16 would also be searched. Therefore, the requirement for restriction should be withdrawn, and rejoinder of claim 16, as amended, is requested.

The invention described and claimed in this application is a finishing composition used in refinishing of vehicles in body shops (see specification page 1, lines 13-19 and page 8, lines 21-27). These compositions are not car waxes or polishes. Finishing compositions are to remove defects in a paint job such as scratch marks and other defects in a vehicle coating. The benefits of this invention are that it: 1. leaves no oily residue (see specification page 1, lines 27-28; page 2, lines 8-15 and 25-26; and page 3, lines 1-2 and lines 21-25; and page 8, lines 28-31); 2. has good handling properties (i.e. acceptable buffing time) see page 8, lines 26-27; 3. is free of non-volatile silicones, droplets of which can contaminate other surfaces (e.g. other cars being painted in a body shop, see specification page 3, lines 3-12); and 4. avoids the phenomenon known as "fish-eye" (see specification page 4, lines 1-4 and page 23, line 4 - page 24, line 15).

The Examiner courteously granted a telephone interview which was held on April 24, 2006. The rejections in the current office action and draft claim amendments were discussed. The remarks which follow reflect the discussion in the interview and are intended to fulfill the requirement for a written statement of the interview.

Claims 12-14 stand rejected under 35 USC § 112, second paragraph, as being indefinite. According to the Examiner's suggestions at pages 3 and 4 of the Office Action, claim 12 as been

amended by canceling the words “effective to create a stable emulsion comprising a volatile cyclic siloxane”, and claim 14 has been amended by replacing “abrasive” with “aluminum oxide” in the penultimate line. In view of these amendments, claims 12 -14 comply with the definiteness requirement of 35USC 112.

Claims 1-5 and 7-8 stand rejected under 35 USC § 102(b) as being anticipated by Kasprzak. This rejection has been avoided by the claim amendments.

Kasprzak is a patent on improvement in polish formulations (see column 1, lines 20-30). Although, the Examiner has correctly pointed out that Kasprzak teaches and claims polishing compositions containing an abrasive, a surfactant (which can be an emulsifier) and a cyclic siloxane, there are substantial differences between Kasprzak’s polish compositions and those of the rejected claims.

1. Kasprzak’s polishes contain polydiorganosiloxane-polyoxyalkylene copolymer, which, being a polymer, is a non-volatile silicone material; whereas, the amended claims require a composition containing less than 0.2 weight percent of non-volatile silicone materials. One of the present inventors, Richard Smith, has informed the undersigned attorney that the polydiorganosiloxane-polyoxyalkylene copolymers of Kasprzak would not be volatile, based on the facts that: the polyoxyalkylene portion has a minimum molecular weight of 1000 and the weight ratio of siloxane portion to polyoxylalkylene portion is in the range of 2-8:1 (col. 1, ll. 52-60) yielding a minimum molecular weight of 3000; and an exemplary polyoxyalkylene (polyethylene oxide) having a molecular weight of 1000 by itself is a non-volatile solid with a melting point of 39°C, and adding polydiorganosiloxane groups would likely further decrease volatility.
2. The Kasprzak compositions do not contain a lubricant. The Examiner has said that the wax specified in Kasprzak reads on lubricant. Claim 1 has been amended to specify a Markush group of lubricants, none of which can be a wax. Waxes are not lubricants, especially the types of waxes found in the cited reference. Kasprzak column 2, lines 11-18 discloses a number of waxes, including beeswax, lanolin, shellac wax, carnauba, bayberry, paraffin and petrolatum, none of which one of skill in the art of making finishing compositions would consider to be a lubricant.

Enclosed with this Response are excerpts from the Kirk-Othmer Encyclopedia of Chemical Technology, volumes 15 and 25, describing lubricant and wax. The process discussed in the Kirk-Othmer Encyclopedia, vol. 15, p. 471 for producing lubricating oil involves the step of removing

paraffin wax. The process for making wax discussed in the Encyclopedia, vol 25, pp. 620-621, shows separation of oil from waxes. Thus, oils and waxes are separated from each other in manufacture and are known in the art as separate classes of materials.

Claims 2, 4, 17 and 18 are further distinguished from Kasprzak. Claim 2 has been amended to specify Markush groups for the volatile siloxane. The volatile siloxane in amended claim 2 is selected from the group identified at specification pages 4 and 5. In claim 4, the volatile siloxane is limited to a Markush group of cyclic siloxanes. In new claim 17, the volatile siloxane is specified as being from a Markush group of siloxanes found in the specification at pages 4 and 5. None of the listed siloxanes are found in Kasprzak, nor are the lubricants. New claim 18 claims the finishing composition of amended claim 1 in which no non-volatile silicone materials have been used in making the composition (based on the examples in the application). Kasprzak uses non-volatile silicones in his composition.

In view of the above differences, Kasprzak does not anticipate claims 1-5, 7-8 and new claims 17 and 18 of the present application.

Claim 6 stands rejected under 35 USC § 103(a) as being unpatentable over Kasprzak in view of Ogawa. This rejection is traversed.

The Examiner has noted Ogawa's recitation of alumina abrasive particles in a finishing composition. However, there are a number of reasons why the combination of Ogawa with Kasprzak does not make claim 6 obvious.

There are reasons related to Ogawa alone. Ogawa teaches the use of alumina particles in non-aqueous finishing agents. The whole Ogawa patent relates to non-aqueous compositions for car finishes and other applications (see abstract, column 1, lines 16-19 and 40-42; column 2, lines 43-46; column 10, lines 44 – column 12, line 25 and claim 1). On the other hand, claim 6 (being dependent from claim 1) requires water in the composition. Any emulsion requires water. Kasprzak requires water, and it is therefore, not reasonable to expect that one would combine Ogawa with Kasprzak.

Ogawa's invention is based on using non-volatile silicones to protect surfaces (column 1, lines 48-51, example 1 and claim 1). Thus, Ogawa teaches away from the present claims which all require that the claimed finishing compositions be substantially free of non-volatile silicone materials.

The differences recited above between Kasprzak and claim 1 also apply to this rejection. In view of the above, claim 6 is not obvious in view Kasprzak combined with Ogawa.

Claims 9-11 stand rejected under 35 USC § 103(a) as being unpatentable over Kasprzak in view of Sejpka et al. This rejection is traversed.

Sejpka discloses a silicone car polish (see abstract, and column 1, lines 10-12 and column 7, lines 65-68). There are substantial differences between the combination of Kasprzak and Sejpka and the rejected claims. The differences already recited above with regard to Kasprzak apply to this rejection.

The Sejpka polishes require the presence of non-volatile silicones (see claim 1 requiring organopolysiloxanes which are solid at room temperature). In contrast, the rejected claims all require that the inventive finishing composition be substantially free of non-volatile silicone materials. Therefore, although both Sejpka and Kasprzak relate to polishes which may be used for cars, combining them would teach a person skilled in the art that he should include a non-volatile silicone material which is contrary to the rejected claims.

Additionally, both references lack disclosure of the lubricant required by amended claim 1, and thus by dependent claims 9-11.

Based on either one of these differences, a *prima facie* case of obviousness does not exist with regard to the amended claims.

Claims 12-13 stand rejected under 35 USC § 103(a) as being unpatentable over Kasprzak in view of Ogawa. This rejection is avoided by the amendments.

Although Kasprzak discloses volatile cyclic siloxanes and surfactants, Ogawa discloses alumina particles, and both patents disclose volatile hydrocarbon solvents, there are significant differences between Kasprzak and Ogawa and claims 12-13. Neither Kasprzak nor Ogawa teaches a finishing composition free of non-volatile silicones. Both patents teach that non-volatile silicone materials should be included in substantial amounts. Kasprzak (column 1, lines 47-60) teaches including 0.5-10% of a polydiorganosiloxane-polyoxy-alkylene copolymer. Example 1 of Kasprzak includes a polydiorganosiloxane-polyoxyalkylene copolymer, and Kasprzak claim 1 requires such materials in his polish. Claim 12, as amended, now requires less than 0.2 weight percent of non-volatile silicone materials.

Ogawa's whole invention is based on using non-volatile silicones to protect surfaces (Ogawa column 1, lines 48-51 and example 1). Ogawa uses the term "finishing agent" in a different sense from the present application. His finishing agent is intended to leave a water-repelling, lustering overcoat chemically bonded to the surface of whatever is being polished (col. 1, ll. 40-47); whereas, the present finishing composition is intended to be applied to a surface to remove scratches and imperfections in a paint job and then be removed.

Neither Kasprzak nor Ogawa teach the use of a non-silicone lubricant as required in claims 12 and 13. Although Kasprzak discloses waxes, they are not lubricants, as discussed above.

In order to arrive at the invention of claims 12 and 13 from the disclosures of Kasprzak and Ogawa, one would have to combine the 2 patents, despite the fact that Kasprzak teaches a water-in-oil polish; whereas, Ogawa's finishing agents are nonaqueous and are not emulsions. Even if one chose to borrow the alumina abrasive from Ogawa and insert it into Kasprzak, one would have to modify the combination by: 1. eliminating the non-volatile silicones (polydiorganosiloxane-polyoxyalkylene copolymers of Kasprzak and organosiloxanes containing a halosilyl group to bond to a surface as taught in Ogawa) from Kasprzak and Ogawa so that the composition could be free of non-volatile silicones; and 2. adding a non-silicone-based lubricant to the composition, despite the lack of any teaching to do so. These modifications are too substantial to be obvious to one of ordinary skill without the hindsight advantage of knowing the present applicant's invention.

Claim 14 stands rejected under 35 USC § 103(a) as being unpatentable over Kasprzak in view of Sejpka et al. This rejection is traversed.

The reasoning applied above to the rejection of claims 9-11 over Kasprzak in view of Sejpka applies to this rejection. Claim 14, being dependent from claim 12, requires a composition containing less than 0.2 percent non-volatile silicones; whereas, both Kasprzak and Sejpka contain non-volatile silicones. Kasprzak's polish formulations contain silicone copolymers, and Sejpka's polishes contain organopolysiloxanes that are solid at room temperature (col. 1, ll. 56-57). Neither Kasprzak nor Sejpka disclose a non-silicone based lubricant, which is required in the finishing compositions of claim 14.

Additionally, claim 14, being dependent from claim 12, requires a volatile hydrocarbon solvent. Sejpka discusses an advantage of his formulations as being solvent free (abstract and col. 8, ll. 1-4). Thus, Sejpka is inconsistent with the formulations covered by claim 14.

There is no reason why one would modify the teachings of Kasprzak and Sejpka to overcome the above recited differences.

Claim 15 stands rejected under 35 USC § 103(a) as being unpatentable over Kasprzak. This rejection has been avoided by the claim amendment.

Kasprzak's polish composition is made by mixing his ingredients under high shear conditions (see Examples 1 and 2). However, his method includes a polydiorganosiloxane-polyoxyalkylene copolymer which is a non-volatile siloxane that would not evaporate. Non-volatile siloxanes are specifically excluded from claim 15. The claim has been amended to say that there is less than 0.2 weight percent non-volatile silicone material used. In addition, claim 15 requires a non-silicone-based lubricant from a Markush group (as now amended); whereas, such a lubricant is missing from the method as taught in Kasprzak. Therefore, the method of claim 15 is not obvious from Kasprzak.

Claims 1-4 and 7-8 stand rejected under 35 USC § 102(b) as being anticipated by Sejpka et al. This rejection has been avoided by the amendments.

Sejpka lacks the following limitations of claims 1-4 and 7-8: being free of non-volatile silicones; and a lubricant selected from the Markush group in amended claim 1. Sejpka requires organopolysiloxanes that are solid at room temperature (abstract, col. 1, ll. 56-57, col. 2, ll. 50-51 and 62-63 and claims). His composition is a silicone car wax (see Example 1). In the present invention, it is desired to remove the composition after the job of eliminating scratches and imperfections from a paint job is done.

Office Action page 9, last paragraph, has equated wax and lubricant, which has been discussed above. Claim 1 has been amended to specify the lubricant more definitely. Sejpka discloses non-silicon-containing waxes at column 6, lines 41-48, such as paraffins, polyethylene waxes, carnauba wax and candelilla wax. None of these would be a suitable lubricant, and none are within the scope of amended claim 1.

Claims 2, 4 and new claims 17 and 18 are further distinguished from Sejpka. Claims 2, 4 and 17 add further limitations to the volatile siloxane that are not found in Sejpka. Claim 18

requires that no non-volatile silicones be used in making the claimed finishing composition, which is below the 0.1% that the examiner has pointed out at Sejpka claim 2.

In view of the above differences, Sejpka does not anticipate claims 1-4 and 7-8, as amended, and new claims 17 and 18.

Claim 5 has been rejected under 35 U.S.C. 103(a) as obvious over Sejpka in view of Kasprzak. This rejection is avoided by the amendments.

The third paragraph at office action page 10, discusses why the Examiner feels the use of volatile hydrocarbon solvent is obvious. This has ignored the recitations in Sejpka (abstract, col. 1, ll. 26-30 and 47-50 and col. 8, ll. 1-4) indicating that his compositions have the benefit of being essentially solvent free. It would not be obvious to deliberately include a volatile hydrocarbon solvent in the inventive finishing composition in view of Sejpka's statements. Since Kasprzak teaches the inclusion of hydrocarbon solvents (col. 2, ll. 28-31 and claim 1) and Sejpka teaches that being solvent-free is an advantage (col. 8, ll. 1-2), it is not obvious to combine them.

Even if one were to combine Sejpka with Kasprzak, he would be missing the lubricant from the Markush group in amended claim 1. Although both Kasprzak and Sejpka disclose various waxes, neither one discloses the lubricants now specified in claim 1 from which claim 5 depends. Hindsight would be required to overcome these differences and arrive at the finishing composition of claim 5.

Claim 6 has been rejected under 35 U.S.C. 103(a) as obvious over Sejpka in view of Ogawa. This rejection is traversed.

Although alumina abrasive is disclosed in Ogawa, there are substantial reasons why the combination of Ogawa and Sejpka does not make claim 6 obvious. As pointed out above, Ogawa relates to non-aqueous compositions; whereas, Sejpka relates to a polish containing water (col. 5, l. 44 and Example 1). There would be no incentive to combine the two patents.

As previously pointed out, Ogawa's invention is based on using non-volatile silicones to protect surfaces (col. 1, ll. 48-51 and example 1). Thus, Ogawa teaches away from claim 6 which requires compositions having less than 0.2 percent non-volatile silicones. Sejpka also contains non-volatile silicones. Also, neither Kasprzak nor Ogawa teach the inclusion of a lubricant, within the class of lubricants in amended claim 1.

Claims 12-13 have been rejected under 35 U.S.C. 103(a) as obvious over Sejpka in view of Ogawa and Kasprzak. This rejection is traversed.

Claims 12 and 13 are limited to finishing compositions containing a volatile cyclic siloxane, a non-silicone-based lubricant and a volatile hydrocarbon solvent.

One would not combine Ogawa with either Sejpka or Kasprzak for the reasons stated above, e.g., Ogawa teaches non-aqueous compositions; whereas both Sejpka and Kasprzak teach compositions containing water in an emulsion.

One would not use Sejpka in combination with Kasprzak because Sejpka teaches the benefit of being solvent-free; whereas, Kasprzak teaches the inclusion of solvents in his polish formulation.

The combination of all three patents, if made, would lack a teaching of a composition free of (containing less than 0.2 weight percent) non-volatile silicone materials, a requirement of claims 12 and 13. All three references include non-volatile silicones in their compositions. In the interview, the Examiner pointed out that, in Sejpka claim 2, the amount of organopolysiloxane is in the range of 0.1 to 5.0%. However, Sejpka also requires that his polish is essentially solvent-free; whereas, claims 12 and 13 required a volatile hydrocarbon solvent. Thus, the skilled person is taught by Sejpka to avoid a formulation containing both non-volatile silicone and solvent, which is a requirement of claim 12.

The combination of these patents would also lack a non-silicone-based lubricant. The waxes in Kasprzak and Sejpka are not lubricants.

Claim 15 has been rejected under 35 U.S.C. 103(a) as obvious over Sejpka. This rejection has been avoided by the claim amendments.

Claim 15, as amended, requires a non-silicone-based lubricant selected from a Markush group; whereas, such an ingredient is missing from the method as taught in Sejpka. Therefore, the method of claim 15 is not obvious from Sejpka.

In view of the above discussion, it is respectfully submitted that claims 1-16, as amended, and new claims 17 and 18 are in condition for allowance. Withdrawal of the restriction requirement and the rejections under 35 U.S.C. 112, 102, and 103 are requested and a notification of allowability is respectfully solicited. If any issues or questions remain the

resolution of which the Examiner feels would be advanced by a conference with Applicants' attorney, he is invited to contact such attorney at the telephone number noted below.

Respectfully submitted,

April 25, 2006
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VOLUME 15

LASERS
TO
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ngs Mountain, N.C.;
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am) 2, 477 (1964).
tad, *Anal. Chem.* 33,

65 (1967).
ille, Okla.
& Latex Co., Akron,

Co., Houston, Tex.
9 (1975).

! (1970).
'81).
ice, Pergamon Press,

'olymerization, Else-

ience and Engineer-

ision, Gastonia, N.C.
Division, Gastonia,

1965).
ntain, N.C.
1034 (1954).

ion, Gastonia, N.C.;
Division, Frankfurt,
astonia, N.C.
nia, N.C.
Lithium in Hexane,

um) 2, 197 (1964).
dam) 4, 95 (1964).
63).
(1963).
64).
, *J. Org. Chem.* 33,

'at. 2,917,539 (Dec.

U.S. Pat. 2,746,900
& Co.).

to the UpJohn Co.).

139. U.S. Pat. 4,039,593 (Aug. 2, 1977), C. W. Kamienski and R. C. Morrison (to Lithium Corp.).
140. U.S. Pat. 3,674,836 (1972), P. L. Creger (to Parke-Davis & Co.).
141. A. S. Kende and P. Fludzinski, *Org. Synth.* 64, 68 (1985).
142. D. J. Hart and co-workers, *J. Org. Chem.* 48, 289 (1983).
143. U.S. Pat. 5,075,439 (1991), F. R. Busch and co-workers (to Pfizer, Inc.).
144. U.S. Pat. 4,320,061 (1982), H. W. Ohlendorf and co-workers (to Kali-Chemie Pharma).
145. *Lithium Methoxide Product Bulletin*, FMC Lithium Division, Gastonia, N.C.; *Lithium Methoxide Product Bulletin*, Chemetall GmbH, Frankfurt, Germany.
146. C. W. Kamienski and D. H. Lewis, *J. Org. Chem.* 30, 3498 (1965).
147. *Lithium t-Butoxide Product Bulletin and Lithium Methoxide Product Bulletin*, Chemetall GmbH, Frankfurt, Germany.

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LITHOGRAPHY. See RESIST MATERIALS.

LUBRICATION AND LUBRICANTS

The primary purpose of lubrication is separation of moving surfaces to minimize friction and wear. Although the fundamental principles were discovered by da Vinci, general understanding of the science of lubrication developed only in the latter part of the nineteenth century (1). Oil film lubrication was discovered in 1885 during studies of railroad car journal bearings in England, and this led almost immediately to the still current theoretical understanding by Reynolds.

Tallow was used to lubricate chariot wheels before 1400 BC. Although vegetable and animal oils were used in following years, significant production of petroleum oils and greases only followed the founding of the modern petroleum industry with the Drake well in Titusville, Pennsylvania in 1859 (2). Production reached 9500 m³/yr (2,500,000 gal/yr) in the following 20 years. Worldwide production is now nearly 1000 times that volume and petroleum lubricants constitute about 98% of total oil and grease production volume.

Lubrication Principles

Several distinct regimes are commonly employed to describe the fundamental principles of lubrication. These range from dry sliding to complete separation of

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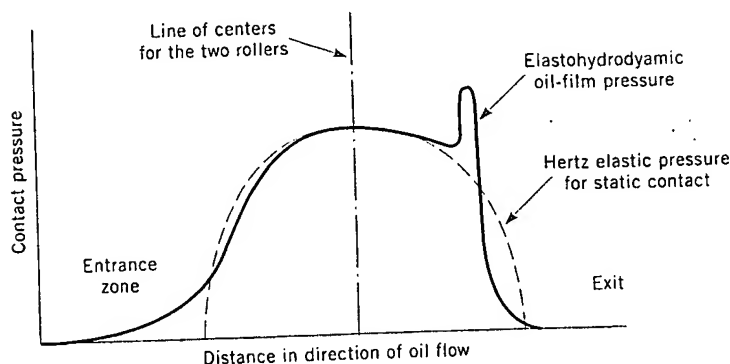


Fig. 5. Pressure distribution between two rollers under load.

In rolling contacts with full separation by an EHL oil film, load capacity is limited primarily by fatigue strength of the metal. Fatigue cracks and spalling under too heavy load are eventually generated by repeated working of grain boundaries about 20–50 μm beneath the contact surface where shear stress is maximum. Surface flaking then occurs with the thickness of loosened particles representing the depth to this zone of maximum shear stress. If the full-film lubrication in the rolling contact is lost under some combination of low speed, high load, low viscosity, or lubricant starvation, increased tangential traction transfers the maximum shear stress out to the metal surface. Surface wear and flaking then occurs (12). In this region of boundary lubrication, lubricant composition and additives may have a pronounced influence, either positive or negative, on fatigue life.

Only very small amounts of oil, less than one drop with most small and medium-sized ball and roller bearings, are sufficient to provide a full EHL film (5). In such cases, a small amount of grease or oil mist balances lubricant loss by vaporization, creepage, and throw-off. With high surface speeds and heavy loads, however, much larger lubricant feed is needed for cooling and makeup.

Petroleum Lubricants

Petroleum (qv) products dominate lubricant production with a 98% share of the market for lubricating oils and greases. While lower cost leads to first consideration of these petroleum lubricants, production of various synthetic lubricants covered later has been expanding to take advantage of special properties such as stability at extreme temperatures, chemical inertness, fire resistance, low toxicity, and environmental compatibility.

Petroleum oils generally range from low viscosity with molecular weights as low as 250 to very viscous lubricants with molecular weights up to about 1000. Typical molecular structures of the complex mixtures of hydrocarbon molecules involved are indicated in Figure 6 (13). Physical properties and performance characteristics depend heavily on the relative distribution of paraffinic, aromatic, and alicyclic (naphthenic) components. For a given molecular size, paraffins have relatively low viscosity, low density, and higher freezing temperatures. Aromatics have higher viscosity, rapid change in viscosity with temperature,

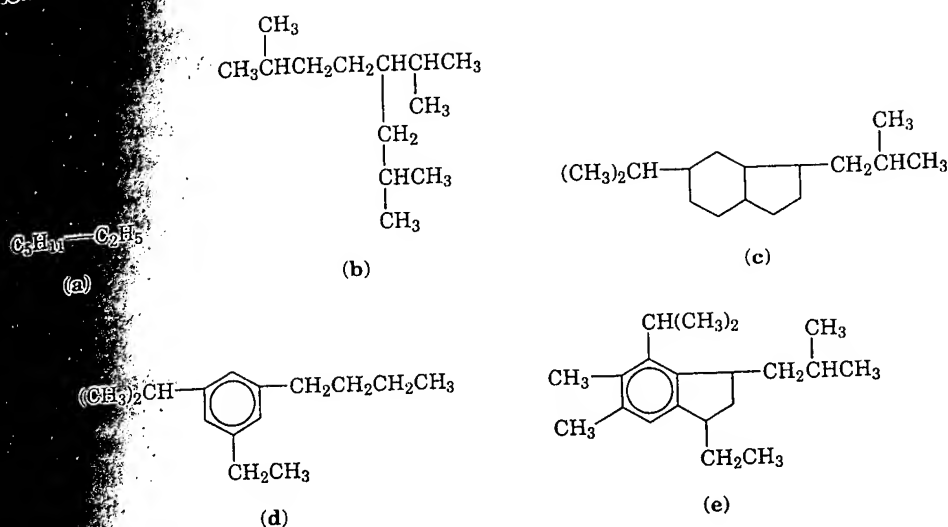


Fig. 6. Typical structures in lube oil: (a) *n*-paraffin, (b) isoparaffin, (c) cycloparaffin, (d) aromatic hydrocarbon, and (e) mixed aliphatic and aromatic ring (13).

higher density, and darker color. Although aromatics have a high degree of oxidation stability, they oxidize to form insoluble black sludge at high temperature. Alicyclic oils are characterized by low pour point, low oxidation stability, and other properties intermediate to those of the paraffins and aromatics.

Almost all premium lubricants are so-called paraffinic oils composed primarily of both paraffinic and alicyclic structures, with only a minor portion of aromatics. When stabilized with an oxidation inhibitor and fortified with other appropriate additives, these paraffinic-alicyclic compositions provide nonsludging oils that are satisfactory for almost any type of service.

The first step in producing a lubricating oil involves distillation (qv) of the crude petroleum (14). The lower boiling gasoline, kerosene, and fuel oils are removed first, and the lubricating oil fractions are then divided by boiling point into several grades of neutral distillates and a final more viscous residuum. Subsequent refining steps remove undesirable aromatics and the minor portion of sulfur, nitrogen, and oxygen compounds. Although solvent extraction or sulfuric acid treatment, followed by activated clay to absorb dark-colored and unstable molecules, had been used for this purification step, hydrogen treatment at high pressure and in the presence of a catalyst was introduced in 1955. Mild hydrofining involves primarily only the removal of color and some nitrogen, oxygen, and sulfur compounds. More severe hydrofining or hydrocracking at temperatures in the 500–575°C range further alters the chemical structures to convert aromatics to paraffins and alicyclics in oils of very high viscosity index (VHVI).

Low temperature filtration (qv) is a common final refining step to remove paraffin wax in order to lower the pour point of the oil (14). As an alternative to traditional filtration aided by a propane or methyl ethyl ketone solvent, catalytic hydrodewaxing cracks the wax molecules which are then removed as lower boiling products. Finished lubricating oils are then made by blending these refined stocks to the desired viscosity, followed by introducing additives needed to

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88. U.S. Pat. 4,661,551 (Apr. 28, 1987), H. Mayer, B. Deubner, P. Iretzberger, R. Muhlhofer, and H. Wilhelm (to Wacker-Chemie GmbH); U.S. Pat. 5,250,106 (Oct. 5, 1993), M. Roth and R. Bernbacher (to Wacker-Chemie GmbH).
89. D. T. Liles and H. V. Lefler III, *Waterborne, Higher-Solids, and Powder Coatings Symposium*, Feb. 1991.
90. U.S. Pat. 5,112,393 (May 12, 1992), J. F. Engel and G. E. Boyer (to ProSoCo, Inc.).
91. U.S. Pat. 4,125,673 (Nov. 14, 1978), M. Roth and H. Gluck (to Wacker-Chemie GmbH).
92. U.S. Pat. 5,442,011 (Aug. 15, 1995), R. A. Halling (to E. I. du Pont de Nemours and Co., Inc.).
93. Eur. Pat. Appl. EP 561,708 (Sept. 22, 1993), L. D. Rich and J. F. Sanders (to 3M Co.).
94. V. M. Malhorta, *Testing Hardened Concrete: Nondestructive Methods*, Iowa State University Press, Ames, Ia., and American Concrete Institute, Detroit, Mich., 1976, pp. 136-137.
95. E. J. Clark, P. G. Cambell, and G. Frohnsdorff, *National Bureau of Standards Technocal Note 833*, National Bureau of Standards, Washington, D.C., Oct. 1975, 79 pp.
96. D. W. Pfeifer and M. J. Scali, *Concrete Sealers for Protection of Bridge Structures*, National Cooperative Highway Research Program Report 244, Transportation Research Board, National Academy of Sciences, Washington, D.C., Dec. 1981.

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WATER GLASS. See SILICON COMPOUNDS.

WATER-SOLUBLE POLYMERS. See Supplement.

WAXES

Wax usually refers to a substance that is a plastic solid at ambient temperature and that, on being subjected to moderately elevated temperatures, becomes a low viscosity liquid. Because it is plastic, wax usually deforms under pressure without the application of heat. The chemical composition of waxes is complex; all of the products have relatively wide molecular weight profiles, with the functionality ranging from products which contain mainly normal alkanes to those which are mixtures of hydrocarbons and reactive functional species.

For centuries, the honeycomb of bees, ie, beeswax, was the material commonly referred to as wax. Substances having typical wax characteristics have traditionally come from insects, eg, beeswax; from vegetables, eg, carnauba; and from animal, eg, spermaceti, origins (1). Waxes from mineral and synthetic sources have been developed both as substitutes for waxes from traditional

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sources and for new applications. Waxes from minerals and synthetic sources now surpass waxes from traditional sources in tonnage and commercial importance. Waxes obtained from natural sources such as vegetables or insects are subject to weather conditions which may severely affect the stability of supply and price and, to a lesser extent, the consistency of the products. Waxes from minerals and synthetic sources are less susceptible to weather conditions, and thus have a more stable supply and price.

Insect and Animal Waxes

Beeswax. White [8012-89-3] and yellow [8006-40-4] beeswax has been known for over 2000 years, especially through its use in the fine arts (2). References to wax prior to the nineteenth century are probably to beeswax. Beeswax is secreted by bees and is used to construct the combs in which bees store their honey. The wax is harvested by removing the honey and melting the comb in boiling water; the melted product is then filtered and cast into cakes. The yellow beeswax cakes can be bleached with oxidizing agents to white beeswax, a product much favored in the cosmetic industry. Imports of beeswax into the United States for the years 1990–1995 are listed in Table 1. Historically, Brazil had long supplied the majority of beeswax to the United States, but now (ca 1997) other countries such as China, Thailand, and Canada supply the majority of the material imported into the United States (3).

The composition of beeswax varies, depending on its geographic origin. The major components are esters of C₃₀ and C₃₂ alcohols with C₁₆ acids, free C₂₅ to C₃₁ carboxylic acids, and C₂₅ to C₃₁ hydrocarbons (4). Beeswax typically has a melting point of 64°C, a penetration (hardness) of 20 dmm at 25°C and 76 dmm at 43.3°C (ASTM D1321), a viscosity of 1470 mm²/s at 98.9°C, an acid number of 20, and a saponification number of 84.

The U.S. Food and Drug Administration (FDA) has affirmed the status of beeswax as Generally Recognized as Safe (GRAS) (5) in Title 21 of the Code of Federal Regulations, Section 184.1973 (21 CFR 184.1973). The major use of beeswax is in the cosmetic industry, with smaller amounts used in pharmaceuticals and candle production.

Spermaceti. Spermaceti [8002-23-1] is derived from the head oil of the sperm whale. Owing to the present status of the sperm whale as an endangered

Table 1. U.S. Imports of Beeswax, Carnauba Wax, Candelilla Wax, and Montan Wax^a

Year	Beeswax		Carnauba wax		Candelilla wax		Montan wax	
	Quantity ^b	Value ^c	Quantity ^b	Value ^c	Quantity ^b	Value ^c	Quantity ^b	Value ^c
1990	1,404	2.49	2,812	2.28	563	2.27	1,664	2.49
1991	823	2.91	3,070	3.65	357	2.49	988	1.71
1992	741	3.02	3,147	3.27	318	2.55	1,369	1.82
1993	901	2.87	3,561	2.30	637	2.61	3,711	0.96
1994	1,111	3.17	2,793	2.70	478	2.65	2,330	1.46
1995	1,382	3.79	3,055	6.08	560	3.03	2,007	1.73

^aRef. 3.

^bQuantity is in metric tons.

^cValue is in U.S. \$ per kg (\$/kg).

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montan wax may be further processed to remove the resins and asphalt, and is known as refined montan wax. White montan wax has been reacted with alcohols to form esters. The wax component of montan is a mixture of long-chain (C_{24} - C_{30}) esters (62-68 wt %), long-chain acids (22-26 wt %), and long-chain alcohols, ketones, and hydrocarbons (7-15 wt %). Crude montan wax from Germany typically has a melting point of 80°C, an acid number of 32, and a saponification number of 92.

The largest traditional use for montan waxes was as a component in one-time hot-melt carbon-paper inks. With the decrease in the use of carbon-paper inks, uses for the refined grades have become predominant, mainly in the formulation of polishes and as plastics lubricants. The alcohol ester derivatives may be used as components of articles intended for use with foods as regulated by the FDA in 21 CFR 178.3770.

Peat Waxes. Peat waxes are much like montan waxes in that they contain three main components: a wax fraction, a resin fraction, and an asphalt fraction. The amount of asphalt in the total yield is influenced strongly by the solvent used in the extraction. Montan waxes contain ca 50 wt % more of the wax fraction than peat waxes, and correspondingly lower percentages of the resin and asphalt fractions. The wax fraction in peat wax is chemically similar to that of the wax fraction in montan wax.

Ozokerite and Ceresin Waxes. Ozokerite wax [8001-75-0] was a product of Poland, Austria, and in the former USSR where it was mined. True ozokerite no longer seems to be an article of commerce, and has been replaced with blends of petroleum-derived paraffin and microcrystalline waxes. These blends are designed to meet the specific physical properties required by the application involved.

Ceresin wax [8001-75-0] originally was a refined and bleached ozokerite wax, but now is a paraffin wax of very narrow molecular weight distribution or blend of petroleum waxes.

Petroleum Waxes. Waxes derived from petroleum are hydrocarbons of three types: paraffin [64742-43-4] (clay-treated); semimicrocrystalline or intermediate; and microcrystalline [64742-42-3] (clay-treated). Semimicrocrystalline waxes are not generally marketed as such (7). Others include acid-treated, chemically neutralized, and hydrotreated; and paraffin and hydrocarbon waxes, untreated. The quality and quantity of the wax separated from the crude oil depends on the source of the crude oil and the degree of refining to which it has been subjected prior to wax separation. Petroleum waxes are produced in massive quantities throughout the world. Subject to the wax content in the crude, paraffin and, to a substantially lesser degree, microcrystalline wax are produced in almost all countries of the world that refine crude oil. Production capacity in the United States and imports for the years 1990 to 1995 are listed in Table 2. Canada supplies over 50% of the petroleum wax imported into the United States (3).

A paraffin wax is a petroleum wax consisting principally of normal alkanes. Microcrystalline wax is a petroleum wax containing substantial proportions of branched and cyclic saturated hydrocarbons, in addition to normal alkanes. Semimicrocrystalline wax contains more branched and cyclic compounds than paraffin wax, but less than microcrystalline. A classification system based on

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Table 2. U.S. Production Capacity and Imports of Petroleum Wax

Year	U.S. Capacity ^a , t	Import Data ^b	
		Quantity, t	Value ^c
1990	585,300	81,306	0.58
1991	551,200	79,176	0.60
1992	624,300	105,297	0.56
1993	614,600	89,330	0.53
1994	634,100	120,599	0.74
1995	643,900	113,800	0.57

^aRef. 3.^bRef. 3.^cValue in U.S. \$ per kg (\$/kg).

the refractive index of the wax and its congealing point as determined by ASTM D938 was developed (9).

Paraffin wax is macrocrystalline, brittle, and is composed of 40–90 wt % normal alkanes, with the remainder C₁₈–C₃₆ isoalkanes and cycloalkanes. Paraffin wax has little affinity for oil content: fully refined paraffin has less than 1 wt %; crude scale, 1–2 wt %, and slack [64742-61-6], above 2 wt %. Within these classes, the melting point of the wax determines the actual grade, with a range of about 46–71°C. Typical properties of petroleum waxes are listed in Table 3.

The separation of paraffin wax from crude oil occurs during distillation, as shown in Figure 1. The distillate is processed to remove oil to the degree desired through solvent extraction. It is then decolorized, usually by hydrogenation, but percolation through bauxite is also used. Microcrystalline wax is produced either from the residual fraction of crude oil distillation or from crude oil tank bottoms (10). After deasphalting of the residual fraction, heavy lubricating oil is removed by solvent extraction. The degree of solvent extraction is dictated by the economics of the lubrication oil market. The filtrate is crude petrolatum, a dark-colored, unctuous material containing oil and microcrystalline wax. Percentages of each may vary, but are usually about 40 wt % wax and 60 wt % oil. This material is then solvent-extracted for the wax. Because microcrystalline wax has great affinity for oil, the oil content of the wax is 1–4 wt %, depending on the grade

Table 3. Typical Properties of Petroleum Waxes

Property	Wax	
	Paraffin	Microcrystalline
flash point, closed cup, °C	204 ^a	260 ^a
viscosity at 98.9°C, mm ² /s	4.2–7.4	10.2–25
melting range, °C	46–68	60–93
refractive index at 98.9°C	1.430–1.433	1.435–1.445
number average molecular weight	350–420	600–800
carbon atoms per molecule	20–36	30–75
ductility/crystallinity of solid wax	Friable to crystalline	Ductile–plastic to tough–brittle

^aValue is minimum.

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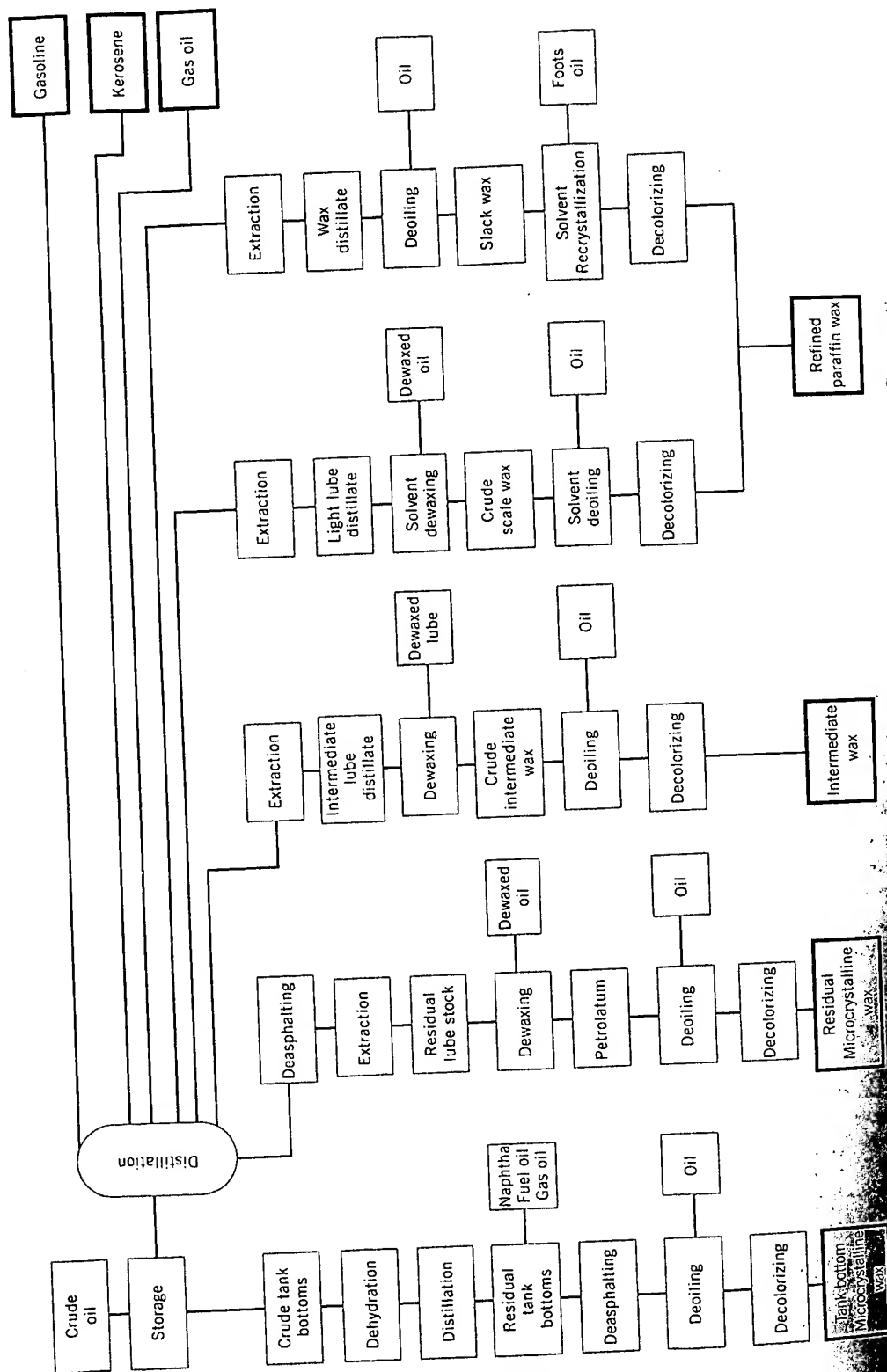


Fig. 1. Refining of petroleum waxes. Courtesy of Baker Petrolite Corporation.

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of the wax. Unlike paraffin wax, oil is held tightly in the crystal lattice of the microcrystalline wax, and does not migrate to the surface. The microcrystalline waxes obtained from petrolatum's are generally known as plastic grades, with penetrations greater than 11 dmm at 25°C.

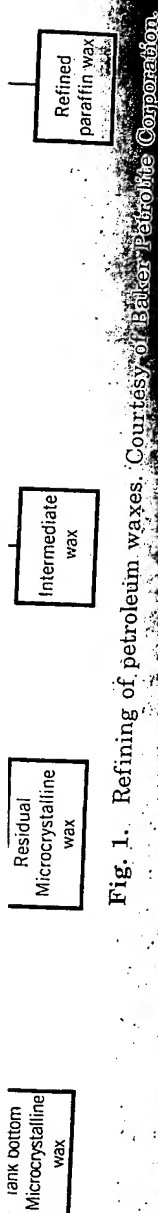
Crude oil contains high molecular weight fractions which are soluble at the high temperatures found in underground formations, but not very soluble at ambient conditions once the crude oil is produced. These high molecular weight fractions precipitate onto the walls and floors of storage tanks, and are known as crude oil tank bottoms. Crude oil tank bottoms are essentially crude oil with very high wax contents and are processed as indicated in Figure 1. The microcrystalline waxes obtained from crude oil tank bottoms are generally known as hard grades, with penetrations less than 11 dmm at 25°C.

The use of refined grades of petroleum waxes in some food applications is regulated by the FDA in 21 CFR 172.886 and 21 CFR 178.3710. The Bundesgesundheitsamt (BGA) of Germany also has specifications for refined petroleum waxes used in food applications. Many other countries reference either the FDA or BGA specifications for their food regulations. Petroleum wax is widely used in chewing gum to modify the properties of the chewing gum base. The wide range of properties available help chewing gum base manufacturers formulate a broad variety of chewing gum, ranging from the traditional hard stick gum to the softer bubble gum. Petroleum wax can also be used as protective coatings for fruits, vegetables, and cheeses. Petroleum wax is outstanding as a cost-effective moisture and gas barrier, and food packaging applications are a major market for refined food-grade petroleum wax. Blends of paraffin and microcrystalline wax are used by themselves or in combination with other additives such as high molecular weight polyethylene and ethylene vinyl acetate copolymers to improve the performance of paper packaging such as paperboard boxes, paper containers, and flexible packaging.

Petroleum waxes are also widely used in other industrial applications. Paraffin waxes are added to rubber during compounding, and exude to the surface during curing, which helps protect the rubber from degradation resulting from ozone. Paraffin and other waxes can be added to plastics, especially poly(vinyl chloride) (PVC) as lubricants. Both paraffin and microcrystalline waxes are widely used to help control the properties of hot-melt adhesives. Dispersions of microcrystalline are added to inks to improve slip and rub properties. Petroleum waxes are used in many consumer applications such as cosmetics, polishes, and candles. Unrefined petroleum waxes are often used in fireplace logs.

Synthetic Waxes

Polyethylene Waxes. Low molecular weight (less than ca 10,000 Mn) polyethylenes [9002-88-4] having waxlike properties are made either by high pressure polymerization or low pressure (Ziegler-type catalysts) polymerization. All the products have the same basic structure, but the processes yield products with distinctly different properties. Some polyethylenes have fairly low densities, owing to branching that occurs during the polymerization. Molecular weight distributions, expressed as the weight average molecular weight divided by the



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